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Kevin -

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Perchlorate testing
Method Question

From: Charles Berrey
To: File

7/15/97

Subject: Phone Messages From Larry Ladd 12:38 AM 7/15

1. In talking to Reber Brown and looking at situations and article I sent you (Enclosure #3 Flow-Injection Extraction-Spectrophotometric Determination of Perchlorate With Brilliant Green) there are any number of old wet chemistry methods to detect perchlorate under 100 ppb. Reber Brown agrees but it was not part of his report he was looking for more efficient rather than old fashion method. As far as ability to detect perchlorate has been around for a long time. <100 ppb.

2. My understanding is that Aerojet's current method was first used in 11/95 sampling with a quantitative limit of 400 ppb and a detection limit of 100 ppb. Using that detection limit have 280 ppb in well # 13 in 1997 and not find perchlorate somewhere in the monitor system at least detection limit in 11/95.

3. Even using Aerojet's old method pick up hit of 2300 ppb at Union Hall well now have 6000 ppb perchlorate. Most recent round hit Jan 30 according to Arden Cordova Water Co. document showed non-detect - someone was finally told to try harder and found perchlorate on 2/11/95. There was no technical breakthrough between 1/30 & 2/11/197.

Ms. Underwood told me the initiative to revise the perchlorate test method in December came as a response to my inquiry.

4. Final item talked to Reber Brown who was not aware that the state Sanitation and Radiation Lab at Berkeley had a perchlorate detected down to 2 ppb. Reber reported back to me that the method was not exotic or expensive.

The critical thing is the ability to detect. The fact sheet said "In the past, the levels at which Aerojet was able to detect perchlorate in the water were much higher than the levels at which there could be some type of health effect. Recently, Aerojet changed to a method which detects perchlorate at much lower levels." Question is ability not whether they was ability or authorized or standardized method. The Technical ability existed and existed for some time to detect perchlorate below 400 ppb or 100 ppb.

The Site Health Assessment meeting has been repeatedly delayed since April.

Duncan Austin
relieved of duty as
w. This a week.

3 between the U.S. De-
ties. N.C. was a visiting
.. Brookfield, CT.

ira. Acta. Part A. 30 (1974).

d Analysis. Wiley. New York

. Instrumentation and Appli-

.) (1977) 261.

Acta. 119 (1980) 189.

4.
381) 201.

: (1980) 734.

al. Chem. 34 (1982) 891.

wet chemistry detection method used
is decades old

Analytica Chimica Acta. 217 (1989) 177-181
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

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Short Communication

FLOW-INJECTION EXTRACTION-SPECTROPHOTOMETRIC DETERMINATION OF PERCHLORATE WITH BRILLIANT GREEN

DOCUMENT 3

D. THORBURN BURNS*, N. CHIMPALEE and M. HARRIOTT

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(Received 13th June 1980)

Summary. Perchlorate ($0-2.5 \mu\text{g ml}^{-1}$) is determined spectrophotometrically at 640 nm after extraction into benzene of its ion-associate with Brilliant Green in a flow-injection manifold with a membrane separator. The injection rate is 20 h^{-1} . The detection limit is 36 ng ml^{-1} based on 20 μl injections. The system is applied to the determination of perchlorate in potassium chlorate after prior selective destruction of chlorate.

36
ppb

Perchlorate can be determined spectrophotometrically after ion-pair liquid/liquid extraction; suitable cations for ion-pair formation reported are methylene blue [1], chelates of copper(I) with cuproin [2] or 6-methylpicolinaldehyde azine [3], tetrabutylphosphonium [4], amiloride (as its hydrochloride) [5] or Brilliant Green [6,7]. Since the introduction of flow-injection liquid/liquid extraction [8,9], numerous phase separators have been developed [10], among the most successful being those based on PTFE membranes [11-13] porous to organic but not to aqueous phases. Gallego and Valcárcel [14] adapted the earlier liquid/liquid extraction method for perchlorate with copper(I)/6-methylpicolinaldehyde azine into MIBK to a flow-injection system, followed by atomic absorption spectrometric measurement. A simpler spectrophotometric method for perchlorate based on the extraction of its ion-pair with Brilliant Green is reported below.

Experimental

Equipment. Absorbances were measured at 640 nm with a Pye Unicam SP 50 ultraviolet-visible spectrophotometer fitted with a 30- μl 10-mm. special optical glass flow cell (Hellma), and recorded with a Phillips PM 8251 recorder. Solutions were pumped with a fixed-speed proportioning pump (Technicon) fitted with Acidflex pump tubes for the organic phase and tygon tubes for the aqueous solutions. Samples were injected from a four-way Rheodyne valve fitted with a by-pass coil. Flow lines were PTFE tubing (0.5 and 0.3 mm i.d.). A diagram of the flow system is shown in Fig. 1. Omnifit three-

January 6, 1997

Ms. Anne Marie Collins
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Answer to 1 university method

RE: Review of Brilliant Green Analytical Method for Analysis of Perchlorate

Dear Anna Marie:

At your request, we have reviewed the analytical method you forwarded to us, "Flow-Injection Extraction-Spectrophotometric Determination of Perchlorate with Brilliant Green," authored by Burns, Chimpalee and Harriott with the Department of Analytical Chemistry, The Queen's University of Belfast in Belfast, Great Britain. The information provided is the result of university research and would require a great deal of additional method development to determine the method's reliability in its application to perchlorate analysis in groundwater.

Telephone

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616.942.6459

Based on an assessment of the method by the director of TriMatrix Laboratories, Inc., Doug Kriscunas, the stated detection limit of 36 ug/L in the published method is probably low by a factor of two to three since it was based on three times the background noise and is not a true method detection limit determined by numerous trials and statistical analysis. The equipment used in the analysis is nonstandard, and would require fabrication. In addition, the method utilizes benzene, which poses potential waste disposal problems and laboratory safety concerns. Finally, the University of Belfast report states that chloride results in positive interference, as do several other ions. The concentration of chloride in the Aerojet groundwater, while low, may be sufficient to create such analytical difficulties. There are a number of other ions present in the Aerojet groundwater which were not assessed for potential interferences by the University, and may also contribute to analytical difficulties.

Please let us know if we can provide any further assistance.

Sincerely,

EARTH TECH, Inc.

Lacy B. Pugh

Lacy B. Pugh, P.E.
Vice President

E A R T H T E C H



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